



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Michihisa TASAKA et al.

Application No.: 09/384,380

Group Art Unit: 1713

Filed: August 27, 1999

Examiner: Rip A. Lee

Confirmation No.: 7724

For: FIRE-RETARDANT RESIN COMPOSITION AND MOLDED PART USING THE  
SAME

DECLARATION UNDER 37 C.F.R. § 1.132

Honorable Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Kazuhiko KOBAYASHI, declare and state that:

1. I am a Japanese citizen residing at 3-26-7, Sugita,  
Isogo-ku, Yokohama-shi, Kanagawa-ken, Japan.

I was graduated from Science & Engineering Section, CHUO  
University in March 1982.

I have been employed by RIKEN VINYL INDUSTRY CO., LTD. (whose  
name is changed to RIKEN TECHNOS CORPORATION as of October 1, 2001)  
since April 1982. I engaged in research and development of  
insulating-materials for electric wires at Compound Technical  
Department of the said company since April 1985. Further, I have  
been engaged in research and development of insulating-materials  
for electric wires in Polymer Application R&D Department at  
Material Research Center of the said company since April 1998.

I am intimately familiar with the contents of United States

Patent Application No. 09/384,380, filed on August 27, 1999, its prosecution before the United States Patent & Trademark Office, and the references cited therein.

2. I have studied the contents of the cited Tasaka et al.'s U.S. Patent No. 6,433,062, and Aida et al.'s U.S. Patent No. 5,221,781.

3. To show the superiority of the present invention, the following tests were conducted, by me or under my supervision:

#### Test

As described in the EXAMPLES section (from line 4 on page 53, to page 72) of the present specification, the resin compositions, sheets of said compositions, and insulated wires having insulating coverings of said compositions of Example 1 and Comparative Examples 1 and 9, respectively, were prepared.

That is, use was made of, as the ingredient (a), a hydrogenated styrene/ethylene/propylene/styrene copolymer (SEPS); as the ingredient (b), a paraffin oil; as the ingredient (c), an ethylene/1-octene copolymer having a density of 0.87 g/cm<sup>3</sup> (c-1); as the ingredient (d), a polypropylene (MFR: 8g/10 min) (d-1) or (d-2), as shown in the following Table B; as the ingredient (e), 2,5-dimethyl-2,5-di(t-butylperoxy)-hexane; as the ingredient (f), triethylene glycol dimethacrylate; and as the component (B), magnesium hydroxide whose surface had been treated with a vinyl silane (B-1) for Example 1 and Comparative example 9, or magnesium hydroxide whose surface had been treated with an

aliphatic acid (B-2) for Comparative Example 1, as shown in Table B, to prepare the compositions, respectively.

In both Example 1 and Comparative Example 1, all of the components were dryblended at room temperature, heated and kneaded in a Banbury mixer at 200 °C, and then discharged at the discharge temperature of 200 °C, to obtain the fire-retardant resin compositions. In Comparative Example 9, after all the components except the metal hydrate (B) were heated and kneaded at 200 °C in a Banbury mixer, the metal hydrate (B) was added, followed by kneading and discharging, to obtain the fire-retardant resin composition. The temperature 200 °C at which the components were heated and kneaded in a Banbury mixer was equal to or higher than the melting temperature of the thermoplastic resin component (A).

The compounds that were used, as shown in Table B, were described in detail in from line 4 on page 61, to the last line on page 64, in particular, the compounds for (B) Metal hydrate are listed again as follows:

(B-1) for Example 1 and Comparative Example 9

Manufacturing company: Kyowa Chemical Co., Ltd.

Trade name: Kisma 5LH

Type: magnesium hydroxide surface-treated with a  
silane coupling agent having a vinyl group at  
the terminal; and

(B-2) for Comparative Example 1

Manufacturing company: Kyowa Chemical Co., Ltd.

Trade name: Kisma 5B

Type: magnesium hydroxide treated with an aliphatic  
acid

This metal hydrate, Kisma 5B (trade name),  $Mg(OH)_2$  all of which was pretreated with an aliphatic acid, is the same one utilized in Examples 15, 16, 17, 18, 20, 21 and 22 of Aida et al.'s U.S. Patent No. 5,221,781 (hereinafter referred to as Aida '781). Please see lines 33 to 36 in column 12, and Table 2 in columns 13 to 16 of Aida '781.

From the thus-obtained resin compositions of Example 1 and Comparative Examples 1 and 9, 1-mm-thick sheets were formed, respectively, as described in lines 11 to 13 on page 55 of the present specification.

In addition, from the resin compositions of Example 1 and Comparative Examples 1 and 9, insulated wires were prepared, respectively, as described in lines 14 to 22 on page 55 of the present specification.

As to the thus-obtained sheets, the tensile properties (extension (elongation) (%) and tensile strength (MPa)) and the heat deformation property were tested and evaluated in the same manner as described in lines 19 to 24 on page 56 of the present specification. The results are shown in Table B.

As to the thus-obtained insulated wires, the tensile properties, the abrasion resistance, the horizontal flame test,

the 60°-inclined flame test, the heat deformation rate test, the whitening test (if a whitening phenomenon was observed when bent), the extrudability test, and the flexibility test were carried out, to test and evaluate the covering layers of each insulated wire in the same manner as described in from line 4 on page 57, to line 3 on page 61 of the present specification. The results are also shown in Table B.

Further, for reference, the resin compositions and results exhibited by the sheets and wires prepared from the compositions, shown in Table A in the Declaration Under 37 C.F.R. § 1.132 dated April 18, 2003, were excerpted and are again shown in Table B below (Comparative example 101 and Example 10). Example 10 contained the metal hydrate (B) {i.e. B-1 and B-2} in a relative amount of 163 parts by weight to 100 parts by weight of the thermoplastic resin component (A) (i.e., {a + b+ c-1 +(d-1 or d-2)}), and more than half the amount of the metal hydrate (B) was made up of Mg(OH)<sub>2</sub> pretreated with a silane coupling agent having a vinyl group at its terminal (B-1). Comparative example 101 contained the same amount of the metal hydrate (B). However, in Comparative example 101, less than half the amount of the metal hydrate (B) was made up of Mg(OH)<sub>2</sub> pretreated with such a silane coupling agent (B-1).

Table B

|  |  | Compara<br>-tive<br>Example<br>1 | Compara<br>-tive<br>Example<br>9 | Example<br>1  | Compara<br>-tive<br>example<br>101 | Example<br>10 |
|--|--|----------------------------------|----------------------------------|---------------|------------------------------------|---------------|
| a  | SEPS   | 100                              | 100                              | 100           | 100                                | 100           |
| b  | Paraffin oil   | 40                               | 40                               | 40            | 40                                 | 40            |
| c-1  | Ethylene/ $\alpha$ -olefin<br>copolymer<br>(ethylene/1-octene<br>copolymer synthesized<br>using single site<br>catalyst)<br>(Density, 0.870) | 133                              | 133                              | 133           | 133                                | 133           |
| d-1  | Block polypropylene  | -                                | -                                | 33            | -                                  | -             |
| d-2  | Random polypropylene   | 33                               | 33                               | -             | 33                                 | 33            |
| e  | Organic peroxide   | 0.66                             | 0.66                             | 0.66          | 0.66                               | 0.66          |
| f  | Crosslinking aid   | 2                                | 2                                | 2             | 2                                  | 2             |
|  | Maleic acid-modified<br>LLDPE  | 27                               | 27                               | 27            | 27                                 | 27            |
| B-1  | Kisma 5LH<br>(Mg(OH) <sub>2</sub> treated with<br>vinyl silane)  | -                                | 500<br>(163)*, *1                | 500<br>(163)* | 200                                | 300           |
| B-2  | Kisma 5B<br>(Mg(OH) <sub>2</sub> treated with<br>aliphatic acid)   | 500<br>(163)*                    | -                                | -             | 300                                | 200           |
|  | Antioxidant  | 3                                | 3                                | 3             | 3                                  | 3             |
|  | Lubricant  | 6                                | 6                                | 6             | 6                                  | 6             |
| Tests of<br>the sheet                              | Extension (%)  | 520                              | 320                              | 200           | 460                                | 390           |
|  | Tensile strength (MPa)   | × 6                              | × 7                              | ○ 19          | × 9.0                              | ○ 12          |
|  | Heat deformation at<br>121 °C (%)  | 20                               | 20                               | 13            | 13                                 | 13            |
| Tests of<br>the<br>insulated<br>(electric)<br>wire | Extension (%)  | 530                              | 170                              | 220           | 430                                | 390           |
|  | Tensile strength (MPa)   | × 6                              | × 7                              | ○ 20          | × 9.3                              | ○ 12          |
|  | Horizontal flame test  | 10/10                            | 10/10                            | 10/10         | 10/10                              | 10/10         |
|  | 60°-inclined flame test  | 10/10                            | 10/10                            | 10/10         | 10/10                              | 10/10         |
|  | Abrasion resistance  | △                                | ○                                | ○             | ○                                  | ○             |
|  | Whitening  | ×                                | ○                                | ○             | ×                                  | ○             |
|  | Heat deformation (%)   | 35                               | 32                               | 21            | 22                                 | 21            |
|  | Extrudability  | ○                                | ○                                | ○             | ○                                  | ○             |
|  | Flexibility  | ○                                | ○                                | ○             | ○                                  | ○             |

Note 1: Each amount of ingredients in the composition is expressed in "parts by weight"

Note 2: "-" means not added

Note 3: \* A numerical value in a bracket shows in a relative amount of Component (B) to 100 parts by weight of the thermoplastic resin Component (A), i.e.  $\{a + b + c - 1 + (d - 1 \text{ or } d - 2)\}$ .

Note 4: \*1 Component (B) was added after heating and kneading at 200 °C the components and ingredients except Component (B).

Note 5: Criteria of evaluation for the properties shown in Table B.

For sheets:

Extension; A value of 100% or more is required to pass the test;

Tensile strength;

10 MPa or more: Good (designated by "O"),

Less than 10 MPa: Not good (designated by "x");

Heat deformation; A value of 30% or less is required to pass the test.

For insulated wires:

Extension; A value of 100% or more is required to pass the test;

Tensile strength;

10 MPa or more: Good (designated by "O"),

Less than 10 MPa: Not good (designated by "x");

Abrasion resistance; The number of movements of the blade was 1000 or more (designated by "O") or 500 or more but less than 1000 (designated by "x"), until in contact with the conductor, each of which passed the test;

Whitening;

No whitening occurrence after winding 6 times: Good (designated by "O"),

Whitening occurrence 6 times or more after winding 6 times: Practically undesirable (designated by "x");

Heat deformation; A value of less than 50% is required to pass the test;

Extrudability;

With a normal load, there is provided an extruded wire-like product having good outer appearance: Acceptable (designated by "O");

Flexibility;

The length of the end lowered from the original level was 3 cm or more: Good (designated "O").

In the results of the horizontal flame test, the numbers of samples that passed the test (per 10 trials) are shown; and in the results of the 60°-inclined flame test, the numbers of samples that passed the test (per 10 trials) are shown.

As is apparent from the results shown in Table B, the sheets prepared from the compositions of Examples 1 and 10 according to the present invention exhibited unexpectedly superior results with respect to tensile strength, compared with the sheets prepared from the compositions of Comparative Examples 1, 9, and 101.

Further, as is apparent from the results shown in Table B, the insulated wires prepared by employing the compositions of Examples 1 and 10 according to the present invention exhibited unexpectedly superior results in tensile strength and whitening, compared with the insulated wires prepared by employing the compositions of Comparative Examples 1, 9, and 101.

The whitening characteristics as mentioned in the above are the properties to be classified into physical properties of a product, such as a wire or a molded plug, as well as aesthetic features thereof. Please note that whitening phenomenon is influenced with flexibility of the product when being bent. If conspicuous whitening occurs to a product, such a product suffering conspicuous whitening may damage an insulating covering made of the resin composition, to cause a serious problem on insulating property of the covering.

In summary, in every test for each of the sheets and the insulated wires, the compositions of the present invention, in which all of the metal hydrate  $Mg(OH)_2$ , or a specific ratio as defined in the present invention of the metal hydrate  $Mg(OH)_2$  was pretreated by a specific silane coupling agent having a vinyl group



or an epoxy group at its terminal, satisfied the criteria required and showed excellent performances. This is in contrast with the poor performances of the compositions of the comparative example, in which, as in Aida '781, all of the metal hydrate  $Mg(OH)_2$  was pretreated by an aliphatic acid, or the metal hydrate  $Mg(OH)_2$  was pretreated with such a silane coupling agent in a ratio but outside the range defined in the present invention.

It is believed that the distinct difference caused between the excellent properties as exhibited in Example 1 and the poor properties as shown in Comparative Example 1 is due to the difference of the surface-treatment (pretreatment) of the metal hydrate either by the specific silane coupling agent having a reactive group such as a vinyl group or an epoxy group at its terminal (in Example 1) or by the aliphatic acid not so reactive as the above silane coupling agent (in Comparative Example 1).

Further, in Comparative Example 9, in which, after the completion of the partial crosslinking reaction, the vinyl silane-treated magnesium hydroxide was added, the effect of improving mechanical properties of electric wires could not be obtained satisfactorily. It can be understood that the effect is not observed unless the metal hydrate is added before or at the same time with the partial crosslinking reaction.

The data already of record in the specification and the supplemental data submitted herewith demonstrate unexpectedly superior results of the claimed fire-retardant resin composition,

molded part, and method for processing fire-retardant resin composition over those of the cited prior art.

4. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: May 7, 2004

Kazuhiko Kobayashi  
Kazuhiko KOBAYASHI